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(54) Title: PIGMENT CONCENTRATE

(57) Abstract

The application relates to a method for preparing a pigment concentrate which is suitable, in particular, for the preparation of a powder coating. The pigment concentrate is prepared by pigment particles being comminuted in the presence of an aqueous binder to form a stable paste or suspension, the said paste or suspension formed being subjected to a drying treatment, and the pigment concentrate being collected. The binder used is preferably free of cosolvent, while expediently it is a resin having a glass transition temperature of from 25° to 55 °C. Preferably, a dispersant is also present. Suitable dispersants are surfactants having an HLB value of from 10 to 18. By using a pigment concentrate formed according to the present method, a powder coating can be formed which no longer has a tinting strength reserve.

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Pigment concentrate.

The invention relates to a method for preparing a pigment concentrate, more in particular to a pigment concentrate suitable for preparing a powder coating.

It should be noted that powder coatings are coating types which are applied by fluidized-bed dip coating or electrostatic spraying. This is followed by baking, as a result of which the powder forms a homogeneous film.

The powder coating is formed by a paint manufacturer by a pigment being mixed with a thermosetting coating system (comprising a resin and a hardener) and additives and/or fillers. The mixture obtained is then homogeneously fused in an extrusion device and is extruded. The extrudate is then cooled to room temperature as rapidly as possible and is finely ground to obtain a powder which can be used as a powder coating.

The drawback of such a powder coating however is that the powder formed may still have a suitable tinting strength reserve, tinting strength reserve being understood as meaning an amount of pigment whose staining power is not utilized.

While said tinting strength reserve can be reduced by the powder obtained being reground to obtain smaller particles, this does lead to higher preparation costs, while this additional processing may not directly lead to the desired end result.

A method for preparing a pigment concentrate suitable for the preparation of a powder coating has now been found, which does not have the abovementioned drawback.

The invention therefore relates to a method of the type mentioned at the outset, which is characterized in that pigment particles are comminuted in the presence of an aqueous binder to form a stable paste or suspension, the paste or suspension formed is subjected to a drying treatment, and the pigment concentrate is collected. The drying treatment applied preferably consists of spray-drying; other drying treatments such as those employing a

drying drum or a belt dryer for example may however also be used.

It should be noted that a stable paste or suspension has been obtained once the comminuted pigment particles no longer settle in the paste or suspension within a specific rest period of the paste or suspension. Usually, a period of 5-30 min. is chosen for this purpose.

During drying, only the amount of water present in the aqueous binder used is evaporated. The present process is therefore particularly environment-friendly, while it is moreover economically advantageous, since no material has to be recovered.

In the method according to the invention preferably, in addition to the binder, a dispersant is also present. A dispersant makes it easier to obtain a stable suspension or paste. The amount of the dispersant to be used is not limited in any particular way; it primarily depends on the pigment type to be used in the concentrate. Normally, however, the pigment concentrate will contain 35-65% of pigment and 4-10% of dispersant, the remainder being (anhydrous) binder.

Expediently, a binder free of cosolvent is employed. In practice it was found, however, that a cosolvent content up to approximately 5% in the binder to be used gives fairly acceptable results. The best results are obtained, however, with a cosolvent content of 0-1%.

In the method according to the invention the binder used is preferably a resin which has a glass transition temperature of from 25 to 55°C. This is because such a material is still found to be readily fusible in the extrusion device during the subsequent formation of the powder coating. It should be noted in this context that the temperature used in the extrusion device is usually from 110 to 130°C.

Binders found to be very expedient for use in the invention are polyester resins, epoxy resins, alkyd resins, polyurethane resins or combinations thereof. An epoxy resin is to be preferred, if the powder coating prepared therewith is to be formed into a coating film which has a

high resistance to chemicals.

Preferably, a saturated polyester resin is used in which hydroxyl groups are present. Such a particularly suitable polyester resin has an acid value between 20 and 55, more in particular between 20 and 30.

It was further found that if the dispersant used is a surfactant having an HLB value of from 10 to 18, preferably from 13 to 17, a pigment concentrate can be obtained which after processing into a powder coating provides a product having very advantageous properties.

It should be noted that the HLB value is a measure for a hydrophilic/lipophilic balance of a substance and indicates how a substance behaves, in total, with respect to water and fat: with a lower HLB value a substance is oleophilic and can no longer be mixed with water; a higher HLB value, in contrast, results in a substance being hydrophilic, which may lead to the powder coating to be prepared being able to be washed off after having been applied to a substrate. The abovementioned HLB values are found to overcome such problems completely.

Preferably, the dispersant used is a nonionogenic surfactant, selected from the group consisting of the ethoxylated fatty alcohols, the ethoxylated nonylphenols and the ethylene oxide/propylene oxide copolymers and block copolymers.

Equally, however, an anionic surface-active phosphate ester or sodium or ammonium salts of poly-electrolytes can be used as the dispersant.

As stated above, in the present invention pigment particles are comminuted with the formation of a stable paste or suspension. It was found in practice that a paste or suspension is stable if a particle size of less than 15 μm is obtained; such a particle size is preferably produced with the use of a bead mill. Obviously, the pigment particles must always be smaller than the thickness of the coating film ultimately to be formed. Moreover, the particle size is important for the gloss of the coating film to be formed, in the sense that the gloss is determined by the particle size of the pigment particles

and their dispersity.

The invention was found to be particular suitable for organic pigments; this is because it is very difficult to disperse such pigments during the manufacture of a powder coating. It was found, however, that as a result of them being used to make a pigment concentrate, such problems can be completely overcome.

Organic pigments which can be used, for example, are carbon black, phthalocyanine green, phthalocyanine blue, organic red and organic yellow.

The invention also relates to a method for forming a powder to be used as a powder coating, by mixing a pigment with a thermoplastic or thermosetting plastic, a hardener and additives, by blending, melting, extruding and finely grinding the mixture into a powder, which method is characterized in that the pigment used is a pigment concentrate prepared according to the method explained above.

The invention further relates to a method for coating a substrate with a powder coating by mixing pigment particles with a thermoplastic or thermosetting plastic, a hardener and additives, melting and extruding the mixture formed, comminuting the extrudate formed into a powder, applying the powder obtained to a substrate in the form of a thin layer and letting said thin layer harden with the formation of a thin film, which is characterized in that the pigment particles used are pigment concentrate particles obtained according to the invention.

The invention is explained below in more detail with reference to the following embodiments.

EXAMPLE I

A mixture was formed from

23.3% by weight of water,

0.2% by weight of anti-foaming agent (NopcoNXZ®, trade name of Henkel AG)[1],

0.1% by weight of preservative (Parmatol A 23®, trade name of Schülke & Mayr GmbH)[1],

2.3% by weight of dispersant (Serad FN 1566®, trade name of Servo for a surfactant)[2],

42.1% by weight of binder in the form of a 43% strength aqueous emulsion of a polyester resin (Uradil, trade name of DSM).

[1] additives, and [2] surfactant comprising an ethoxylated nonylphenol having an HLB value of 13.5.

The mixture formed was admixed with 32.0% by weight of chromophthal blue ABR, the said pigment being dispersed with the aid of a bead mill to a maximum particle size of 10 microns. The paste was then sprayed, and the desired pigment concentrate particles were obtained which consisted of 61.0% by weight of pigment, 34.6% by weight of binder and 4.4% by weight of surfactant.

Processing the pigment concentrate formed to give a blue powder coating gave a powder coating having good hiding power and a gloss as desired, while the desired tinting was obtained directly over the entire substrate to be coated.

EXAMPLE II

Using the same substances as in Example I, a mixture was formed from:

26.4% by weight of water,
0.3% by weight of anti-foaming agent,
0.1% by weight of preservative,
2.8% by weight of surfactant, and
40.2% by weight of binder emulsion.

This mixture was admixed with 30.2% by weight of PV-Echtgelb HG, the said pigment being dispersed with the aid of a bead mill to a maximum particle size of 10 microns in the form of a stable paste. The paste was sprayed, and pigment concentrate particles were obtained which consisted of 60.0% by weight of pigment, 34.4% by weight of binder and 5.6% by weight of surfactant.

In the course of the pigment concentrate particles obtained being processed into a yellow powder coating for industrial applications, the pigment concentrate particles were found to be eminently compatible with the raw materials customary for a powder coating.

Furthermore it was found that the powder coating thus formed no longer had a tinting strength reserve.

EXAMPLE III

Using the same substances as in Example I, a mixture was formed from:

33.0% by weight of water,
0.3% by weight of anti-foaming agent,
0.1% by weight of preservative,
4.2% by weight of surfactant, and
34.1% by weight of binder emulsion.

This mixture was admixed with 28.3% by weight of PV-Rot HG, the said pigment being dispersed with the aid of a bead mill to a maximum particle size of 10 microns to obtain a stable paste. The paste was subjected to spray-drying, and pigment concentrate particles were obtained which consisted of 60.0% by weight of pigment, 31.2% by weight of binder and 8.8% by weight of surfactant.

Red powder coating formed by using the pigment concentrate particles obtained had excellent characteristics.

EXAMPLE IV

Using the same substances as in Example I, a mixture was formed from:

18.2% by weight of water,
0.3% by weight of anti-foaming agent,
0.1% by weight of preservative,
3.5% by weight of surfactant, and
39.7% by weight of binder emulsion.

The mixture formed was admixed with 38.2% by weight of Phthalogreen 3G 220, the said pigment being dispersed with the aid of a bead mill to a maximum particle size of 10 microns to form a stable paste. The paste was sprayed, and pigment concentrate particles were obtained which consisted of 65.0% by weight of pigment, 29.1% by weight of binder and 5.9% by weight of surfactant.

Green powder coating formed by using the pigment concentrate particles obtained had excellent characteristics.

EXAMPLE V

Using the same substances as in Example I, a mixture was formed from:

28.6% by weight of water,
0.3% by weight of anti-foaming agent,
0.1% by weight of preservative,
4.3% by weight of surfactant, and
36.6% by weight of binder emulsion.

The mixture formed was admixed with 30.1% by weight of Printex 75, the said pigment being dispersed with the aid of a bead mill to a maximum particle size of 10 microns, to obtain a stable paste. The paste was sprayed, and pigment concentrate particles were obtained which consisted of 60.0% by weight of pigment, 31.3% by weight of binder and 8.7% by weight of surfactant.

The pigment concentrate particles thus formed were found to be eminently compatible with the raw materials customary for preparing a (black) powder coating.

EXAMPLE VI

A mixture was formed from
37.2% by weight of water,
additives consisting of:
0.2% by weight of anti-foaming agent (Nopco NXZ), and
0.1% by weight of preservative,
dispersant consisting of:
2.7% by weight of phosphate ester (Serad FA 192, trade name of Servo), and
0.3% by weight of sodium salt of a polyelectrolyte (Orotan 731, trade name of Röhm GmbH)
and
22.4% by weight of binder, consisting of a 90% strength aqueous emulsion of a polyurethane polyol (K-Flex UD 320 W, trade name of King Industries).

This mixture was admixed with 37.3% by weight of Heliogen blue K 6911 D (trade name of BASF), the said pigment being dispersed in a bead mill to a maximum particle size of 10 microns to form a stable paste. The paste was sprayed, and pigment concentrate particles having the same favourable characteristics as the concentrate particles prepared in the previous examples were obtained.

It should be noted that for comparison purposes the binder used was an aqueous emulsion of a non-drying alkyd

resin and an oil-free alkyd resin instead of the polyester resin and polyurethane polyol employed in the previous examples. Such emulsions are commercially distributed, for example, by Worlee under the designation Worleesol 84 (a non-drying alkyd resin with a dry substance content of 44%) and Worleepol V450 and Worleepol 808 (both oil-free alkyd resins with dry substance contents of 90% and 98%, respectively). The results which were obtained with the pigment concentrates thus formed were completely comparable with the concentrates obtained on the basis of a polyester resin or a polyurethane polyol.

CLAIMS

1. Method for preparing a pigment concentrate, suitable for preparing a powder coating, **characterized in that** pigment particles are comminuted in the presence of an aqueous binder to form a stable paste or suspension, the paste or suspension formed is subjected to a drying treatment, and the pigment concentrate is collected.

2. Method according to claim 1, **characterized in that** in addition to the binder a dispersant is also present.

3. Method according to claim 1 or 2, **characterized in that** a binder having a cosolvent content of 0-5% is used.

4. Method according to one or more of the claims 1 to 3, **characterized in that** the binder used is a resin having a glass transition temperature of 25-55°C.

5. Method according to one or more of the claims 1 to 4, **characterized in that** the binder used is a polyester resin, an epoxy resin, an alkyd resin, a polyurethane resin or a combination thereof.

6. Method according to claim 4 or 5, **characterized in that** a saturated polyester resin having an acid value of from 20 to 55, preferably 20-30, is used.

7. Method according to claim 2, **characterized in that** the dispersant used is a surfactant having an HLB value of from 10 to 18, preferably from 13 to 17.

8. Method according to Claim 2 or 7, **characterized in that** the dispersant used is a nonionic surfactant, selected from the group consisting of the ethoxylated fatty alcohols, the ethoxylated nonylphenols and the ethylene oxide/propylene oxide copolymers and block copolymers.

9. Method according to claim 2 or 7, **characterized in that** the dispersant used is an anionic surface-active phosphate ester or a sodium or ammonium salt of a polyelectrolyte.

10. Method according to one or more of the claims 1 to 9, **characterized in that** the pigment particles are comminuted, with the use of a bead mill, to a particle size of less than 15 μm.

11. Method according to one or more of the claims 1 to

10, characterized in that the pigment used is an organic pigment, in particular selected from carbon black, phthalocyanine green, phthalocyanine blue, organic red and organic yellow.

12. Pigment concentrate obtained with the use of the method according to one or more of the claims 1 to 11.

13. Method for forming a powder to be used as a powder coating, by mixing a pigment with a thermoplastic or thermosetting plastic, a hardener and additives, by blending, melting, extruding and finely grinding the mixture into a powder, characterized in that the pigment used is a pigment concentrate according to claim 12.

14. Method for coating a substrate with a powder coating by mixing pigment particles with a thermoplastic or thermosetting plastic, a hardener and additives, melting and extruding the mixture formed, comminuting the extrudate formed into a powder, applying the powder obtained to a substrate in the form of a thin layer and letting said thin layer harden with the formation of a thin film, characterized in that the pigment particles used are pigment concentrate particles obtained according to the method according to one or more of claims 1 to 11.

INTERNATIONAL SEARCH REPORT

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A. CLASSIFICATION OF SUBJECT MATTER
IPC 6 C09B67/20 C09D5/03 C09C1/56

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
IPC 6 C09B C09D C09C C08J

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C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category *	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	CH-A-485 007 (ICI) 13 March 1970 see the whole document	1-14
Y	US-A-3 759 864 (P.N. NICKS) 18 September 1973 see abstract; examples	1-14
A	FR-A-2 311 826 (BAYER) 17 December 1976 see page 3, line 9 - line 18	1-14
A	US-A-4 168 180 (A.R.PEABODY) 18 September 1979 see abstract	1-14
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information on patent family members

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